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SUMMARY

Tensile creep studies were conducted on polycrystalline Nextel 610 and Fiber FP alumina fibers with grain sizes of 100 and 300 nm, respectively. Test conditions were temperatures from 800 to 1050 °C and stresses from 60 to 1000 MPa. For both fibers, only a small primary creep portion occurred followed by steady-state creep. The stress exponents for steady-state creep of Nextel 610 and Fiber FP were found to be about 3 and 1, respectively. At lower temperatures, below 1000 °C, the finer grained Nextel 610 had a much higher 0.2 percent creep strength for 100 hr than the Fiber FP; while at higher temperatures, Nextel 610 had a comparable creep strength to the Fiber FP. The stress and grain size dependencies suggest Nextel 610 and Fiber FP creep rates are due to grain boundary sliding controlled by interface reaction and Nabarro-Herring mechanisms, respectively.

INTRODUCTION

High temperature structural applications, such as, advanced gas turbine engines for civil transport aircraft, have generated great interest in intermetallic and ceramic composites reinforced by high strength continuous length fibers. The use temperatures depend upon a variety of fiber properties, such as, oxidation resistance and creep. Because of their composition, alumina fibers can withstand extremely high temperatures in an oxidizing atmosphere (ref. 1). However, in terms of creep behavior, fine diameter polycrystalline alumina fibers (Fiber FP and PRD 166) with grain sizes of ~500 nm have recently been shown to possess low tensile creep strengths of only 50 MPa at 1150 °C for 0.2 percent creep strain in 5 hr (ref. 2). At this temperature, grain boundary diffusion and interface reaction creep were reported to control the creep properties. Recently a more finely grained polycrystalline alumina fiber, Nextel 610, has been developed by the 3M Company. Because of its finer grain size (~100 nm), this fiber has a very high room temperature tensile strength (ref. 3), but may creep more than previously available alumina fibers.

The primary objective of this study was to measure the tensile creep behavior of two polycrystalline oxide fibers, Nextel 610 and Fiber FP, which have measurably different grain sizes. The purpose was to understand whether the new Nextel fiber with improved room temperature strength offered any creep strength advantages over the older FP fiber. Emphasis was placed on the low strain region below 1 percent since this value is often quoted as the upper creep limit for structural composites. In addition, an analysis was performed to identify the underlying mechanisms, especially in regards to the effects of grain size.

EXPERIMENTAL PROCEDURE

As indicated in table I, the compositions of the fibers used in this study were more than 99 percent alumina with additions of SiO_2 and Fe_2O_3 for Nextel 610 and MgO for Fiber FP. These additives serve as aids to consolidation and as microstructural control agents (in particular against grain growth). The diameters of these fibers were nominally 14 and 20 μm for the Nextel 610 and Fiber FP, respectively. Average grain sizes were 100 and 300 nm for the Nextel 610 and Fiber FP, respectively. Grain sizes were measured using transmission electron microscopy (TEM) along with the line intercept method (about 50

grains were counted on TEM micrographs). TEM micrographs of the as-received Nextel 610 and Fiber FP, shown in figure 1, reveal equiaxed, densely packed grains with no observable amorphous phase.

For this investigation tensile creep measurements were made by dead weight loading. The test temperatures were in the range of 800 to 1050 °C with loading up to 100 hr. The tests were done mostly in air, but some experiments were done in a vacuum of 10^{-5} Pa with no observable difference in the results.

In performing the elevated temperature tests, hot grips were used (fig. 2). The grip was constructed by using a 125- μ m sapphire fiber as top and bottom support wires and securing the fiber specimen with a high temperature zirconia base cement (Sauereisen Cements Company, Pittsburgh, PA). This cement did not interact with the specimen nor did it allow slippage at the temperatures and stress levels used in this investigation. Small diameter (tungsten) wire hooks were used to minimize bending moments in the load train. The specimen's gage length between the cemented ends was 25 mm. The fiber deformation was monitored by a free floating cored LVDT (Schaevitz Engineering, Pennsauken, NJ). The displacement sensor was interfaced with a computer to record the data. The furnace employed for this study was a Pt wire wound tube furnace which allowed continuous operation in air up to 1150 °C with a hot zone length of about 25 mm. The maximum gradient along the hot zone was within 6 °C. A Pt-Rh thermocouple and electronic controller provided a constant temperature in the hot zone of ± 2 °C.

RESULTS

Typical results for fiber creep deformation versus time for the Nextel 610 and Fiber FP are shown in figure 3. In this figure, creep data were taken from 900 to 1050 °C in vacuum and are compared with Fiber FP data obtained from Pysher et al. (ref. 2) at 1150 °C in air. These data, which were obtained by using hot grips with 25 mm long specimens, are in good agreement with Pysher's values which were obtained by cold grip techniques with about 350 mm long specimens. For both fibers, only a small primary creep portion occurred, followed by steady state creep which dominated the deformation at each temperature. In general, the FP fiber required lower stress than Nextel 610 to obtain the same order of creep strain. At temperatures of 900 and 980 °C, and times up to 40 hr, creep deformations ranged from 0.1 to 0.4 percent; while at the higher temperature of 1050 °C, deformations on the order of 1 percent were observed in 4 hr.

Figure 4 illustrates the temperature dependence of the steady state creep rates of (a) Nextel 610 and (b) Fiber FP. In general, at low stresses, Nextel 610 had a lower creep rate than Fiber FP, but a comparable creep rate at high stresses, above ~ 300 MPa at 980 °C and ~ 500 MPa at 900 °C. Least squares fit lines through the data in figure 4 suggest a power law creep relation of $\dot{\epsilon} \approx \sigma^n$. From the best fit lines, the stress exponents n were determined to be 2 to 3 for the Nextel 610 and about 1 for Fiber FP. It can also be seen that the data exhibit an increase in scatter with decreasing temperature.

Figure 5 illustrates the effects of temperature and grain size on the steady state creep rates of polycrystalline alumina material for a stress of 410 MPa and temperatures in the range of 800 to 1600 °C. The steady-state creep rates of the Nextel 610 and Fiber FP from 800 to 1050 °C were determined from figure 4. Data above 1050 °C in figure 5 were from Pysher et al. (ref. 2) for Fiber FP and from Heuer et al. (ref. 4) for bulk alumina. These last data were extrapolated to 410 MPa for comparison with the fibers. Based on best fit lines through the data, the creep rate of the Nextel 610 appeared to be higher than that of Fiber FP and bulk alumina at higher temperatures. However, at lower temperatures below about 1000 °C, Nextel 610 possessed a comparable creep rate to the Fiber FP. A possible reason for this is a higher stress exponent for Nextel 610 than Fiber FP.

The apparent activation energy for creep, Q_{creep} , can be obtained from the slope of the best fit line using the following equation:

$$Q_{\text{creep}} = R (\Delta \ln(\dot{\epsilon}) / [\Delta(1/T)]) \quad (1)$$

where R is the universal gas constant, $\dot{\epsilon}$ is the creep rate (s^{-1}), and T is the absolute temperature (K). The Q_{creep} was determined to be about 460 kJ/mol for both Nextel 610 and Fiber FP from 800 to 1050 °C. This value is somewhat lower than that of Pysher et al. (ref. 2) who reported to be 588 kJ/mol for Fiber FP at 100 MPa. On the other hand, the 460 kJ/mol value from this study appears to agree with the bulk alumina data and is similar to that of Al cation activation energy for diffusion in bulk alumina at high temperature (ref. 5).

The stress levels needed to obtain a creep deformation of 0.2 percent for times up to 100 hr are shown as a function of temperature in figure 6 for (a) Nextel 610 and (b) Fiber FP. At 900 °C, the 0.2 percent creep strength of Nextel 610 is much higher than that of Fiber FP for 10 and 100 hr, but at higher temperature the difference in strength decreases. The slope in figure 6, that is, the required stress difference between 10 and 100 hr for 0.2 percent creep strain, increases with increasing temperature. This may be due in part to 0.2 percent creep occurring in different creep stages, such as, the late stage of steady-state creep at lower temperatures and the early stage of steady-state creep at higher temperatures.

TEM micrographs of the creep tested Nextel 610 and Fiber FP are shown in figure 7. Equiaxed, densely packed initial grains remained unchanged, suggesting a stable fine grain microstructure during creep testing with no significant grain growth. At about 1 percent creep strain and the onset of the tertiary creep region for Nextel 610, the TEM micrographs revealed no grain elongation to the tensile stress axis and no triple-point fold or cavitation along the grain boundaries. Some void-like features in the grains are believed due to an artificial effect formed during specimen preparation.

DISCUSSION

The single filament hot-grip creep results of this study were found to be comparable with literature creep data measured on similar fiber specimens that were cold gripped outside of the furnace hot zone (refs. 2 and 3). This hot-grip technique can be especially advantageous for high temperature tensile studies in which only a short fiber is available. However this method needs to be further evaluated at higher temperatures in which the zirconia-based cement may have adhesion limits. Also, for high stresses and extremely brittle and stiff fibers, bending moments between the support wire and specimen may cause premature fracture.

The relatively low temperature creep deformation with stress exponents of 3 to 1 for the Nextel 610 and Fiber FP fibers is believed to be related to grain boundary sliding controlled by grain boundary diffusion. In general, the steady-state creep rate, $\dot{\epsilon}$, can be expressed in terms of the tensile stress, σ , absolute temperature, T , and grain size, d , by the relation:

$$\dot{\epsilon} = A \sigma^n (-Q_{\text{creep}}/RT) d^{-p} \quad (2)$$

Here n is the stress exponent, p the grain size exponent, Q_{creep} the activation energy for creep, and A the creep constant. Rachinger (ref. 6), and Cannon and Langdon (ref. 7) have analyzed the creep deformation of polycrystalline materials with various creep mechanisms among which are grain boundary sliding controlled by various diffusion mechanisms. These mechanisms differ by their characteristic n and p values. In grain boundary sliding, stress exponents of $n = 1$ are usually associated with free condensation

of mobile vacancies at the grain boundaries with no diffusion limits (Nabarro-Herring or Coble creep). However in the case of grain boundary sliding controlled by interface reaction diffusion, the mobility of the vacancies are hindered, possibly by stress fields in the lattice induced by substitution or interstitial atomic point defects, thereby resulting in $n = 2$.

Creep exponents and parameters for the two fibers are summarized in table II. The creep constant, A , was determined assuming that the grain size influence (d^{-p} in eq. (2)) is not known. For Fiber FP, the low stress exponent of $n \sim 1$, which gives a rise to a higher A constant, suggests creep by grain boundary sliding controlled by unlimited diffusion. For Nextel 610, the stress exponent of $n \sim 3$ suggests interface reaction controlled grain boundary sliding which may be diffusion limited by the processing additives silica and hematite. Silica as a glass former might cause viscous flow, but this would result in creep exponent of unity (ref. 7). Also no residual amorphous phase is found in the grain boundaries of the Nextel 610. Although the observance of a stress exponent of greater than the unity is not clear at this time, it may be due to the combined effect of the additives. In Nextel 610 the hematite has the same crystal structure as alumina and preserves electrical neutrality; hence little misfit strain nor defects are expected. However the silica with its tetravalent cation silicon may cause complex defects which have higher activation energies for mobility than those created by doping with divalent cations, such as, magnesium in the case of Fiber FP.

In general, ultra fine grained ceramic materials are more susceptible to creep deformation than coarse grained materials. Creep rates increase as an inverse power, p , of grain size. Based on the tendency of figure 5 at 1200 °C, the Nextel 610 has the highest creep rate, and the 1 to 3 μm bulk alumina the lowest creep rate. The grain size of the polycrystalline alumina fibers and bulk alumina appear to effect the steady-state creep rate by $p \sim 2$, as shown in figure 8. Based on creep theories (refs. 6 and 7) this may be related with Nabarro-Herring type of creep, Rachinger sliding with a formation of triple-point fold formation, or interface reaction controlled creep. Based on $n \sim 1$ for Fiber FP, it would appear that the creep mechanism for the Fiber FP is grain boundary sliding controlled by Nabarro-Herring creep. On the other hand, for Nextel 610, the observance of $n \sim 3$ and $p \sim 2$ suggests that at least at low temperatures, the creep mechanism is grain boundary sliding controlled by interface reaction creep. The comparable creep rate of the Nextel 610 to that of the Fiber FP from 800 to 1050 °C might then be explained in part by reduced Nextel 610 creep due to the interface reaction mechanism.

CONCLUSIONS

For times up to 100 hr, Nextel 610 has greater 0.2 percent creep strength below 1000 °C than Fiber FP. The greater creep strength of Nextel 610 appears to be related to a higher stress dependence for creep rate which may in turn be related to smaller grain size and/or impurity content. The stress and grain size dependencies suggest Nextel 610 and Fiber FP creep rates are due to grain boundary sliding controlled by interface reaction and Nabarro-Herring mechanisms, respectively.

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TABLE I.—NOMINAL PROPERTIES OF
POLYCRYSTALLINE ALUMINA FIBERS

Property	Nextel 610	Fiber FP
Manufacturer	3M	Dupont
Composition, wt. %	>99% α -Al ₂ O ₃	>99% α -Al ₂ O ₃
Impurities, wt. %	0.2 to 0.3 SiO ₂ 0.4 to 0.7 Fe ₂ O ₃	~0.3 MgO
Average diameter, μ m	14	20
Elastic modulus at RT, GPa	~380	~380
Average grain size, nm	100	300

TABLE II.—STEADY-STATE CREEP PARAMETERS FOR
NEXTTEL 610 AND FIBER FP ALUMINA FIBERS

Fiber	A	Q	p	n	Possible creep mechanism
Nextel 610	2.1×10^4	~460	2	~3	Interface reaction
Fiber FP	4.9×10^9	kJ/mol	2	~1	Nabarro-Herring or Coble

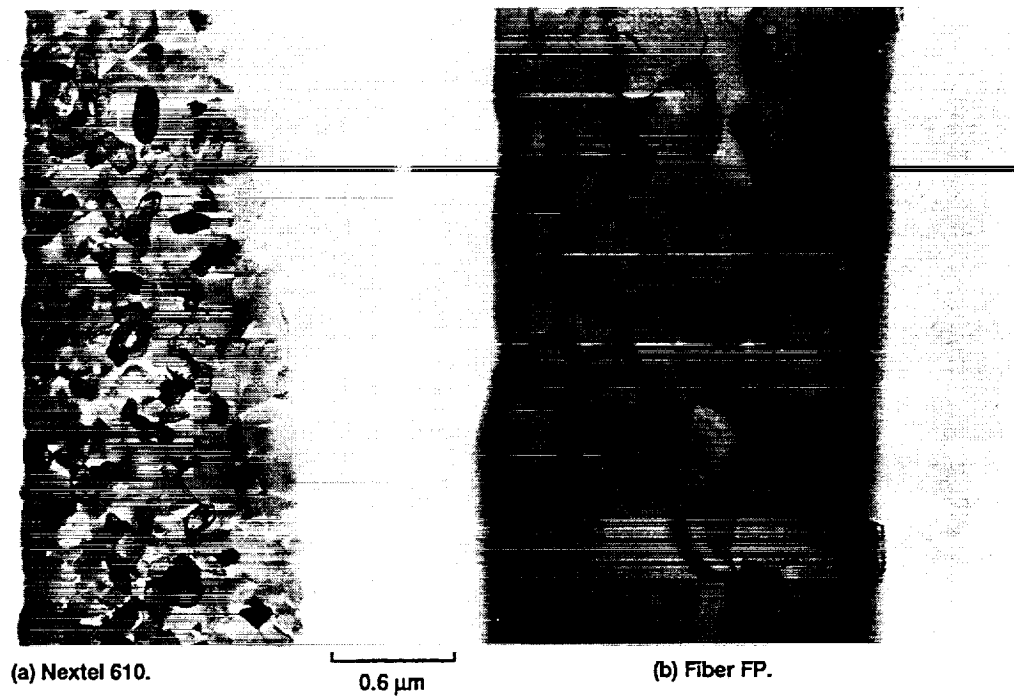


Figure 1.—TEM micrographs of as-received polycrystalline (a) Nextel 610 and (b) Fiber FP, longitudinal sections.

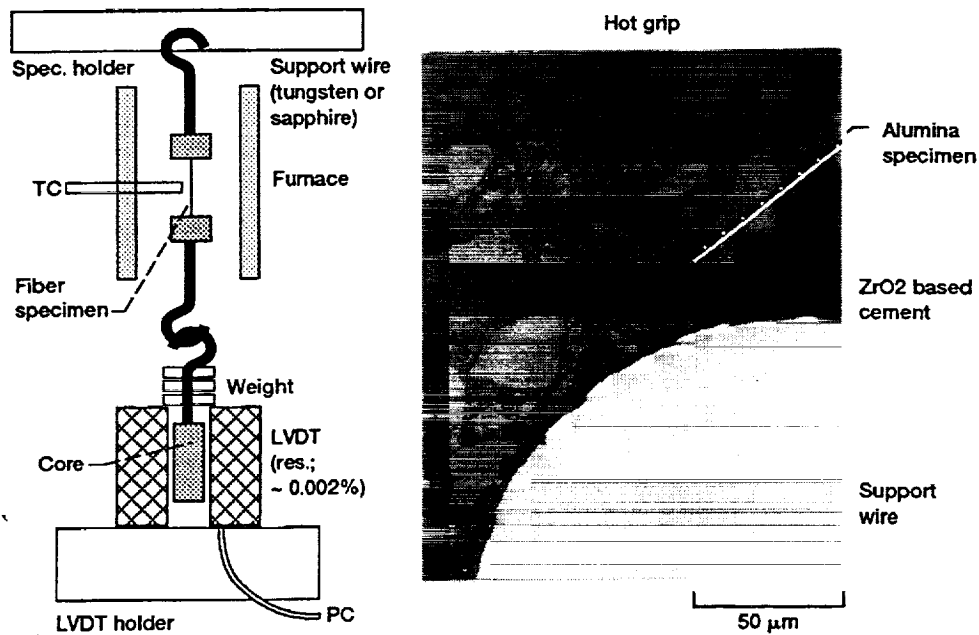


Figure 2.—Single filament creep test rig (gauge length of 25 mm).

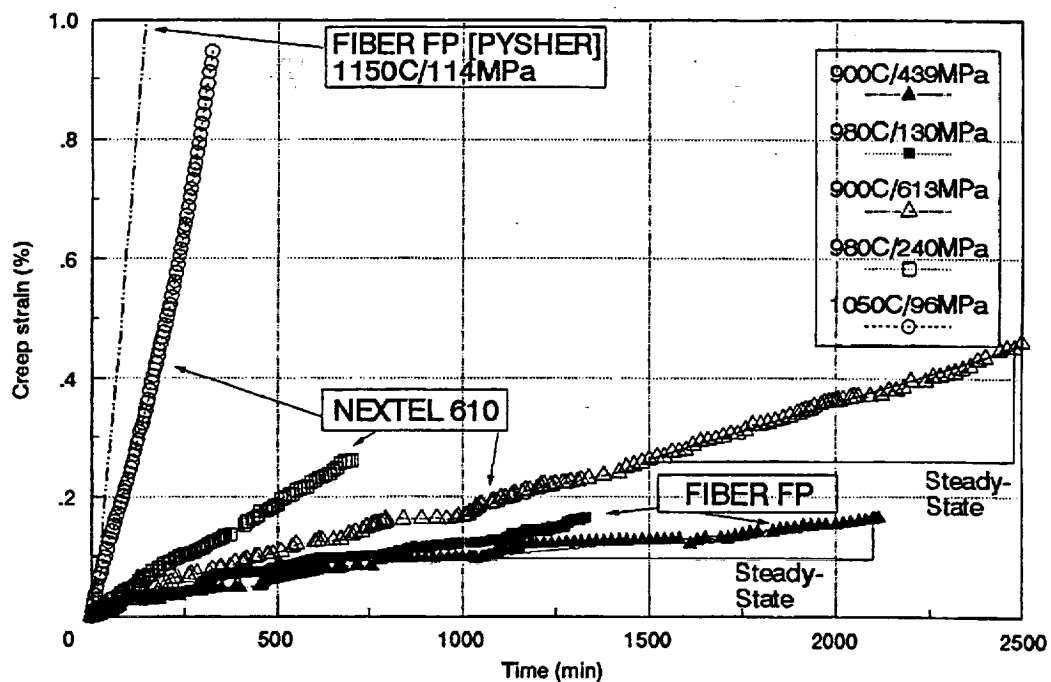


Figure 3.—Typical creep curves of Nextel 610 and Fiber FP fibers.

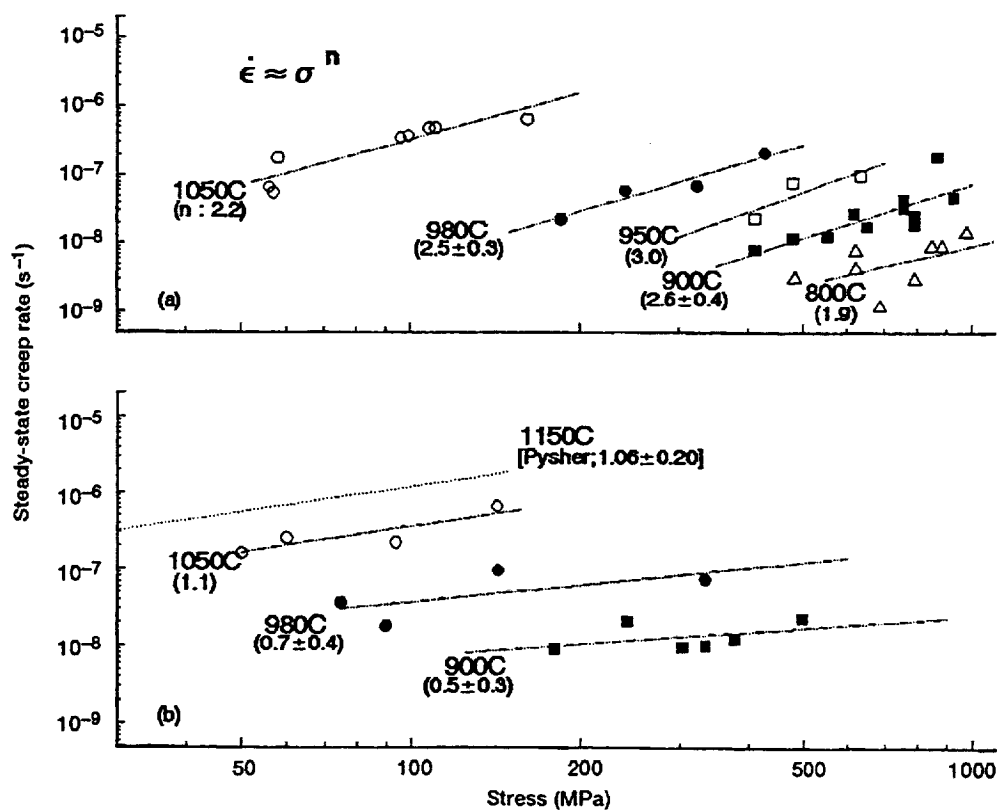


Figure 4.—Stress-effects on steady-state creep rate of (a) Nextel 610 and (b) Fiber FP.

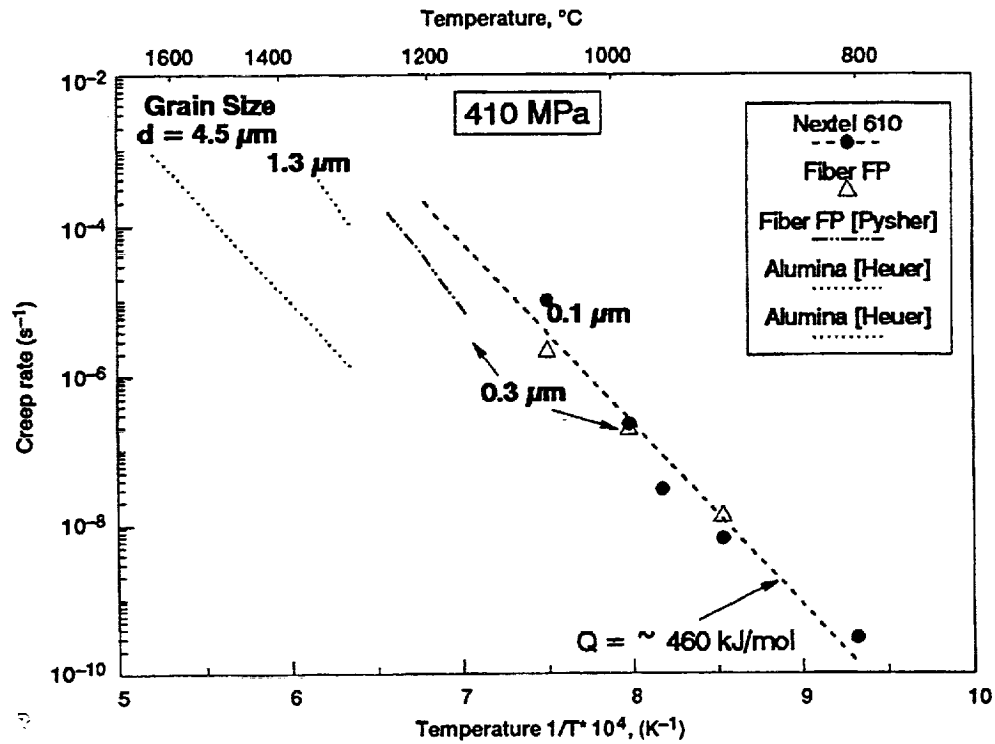


Figure 5.—Temperature effects on the creep behavior of polycrystalline alumina fibers compared to bulk alumina.

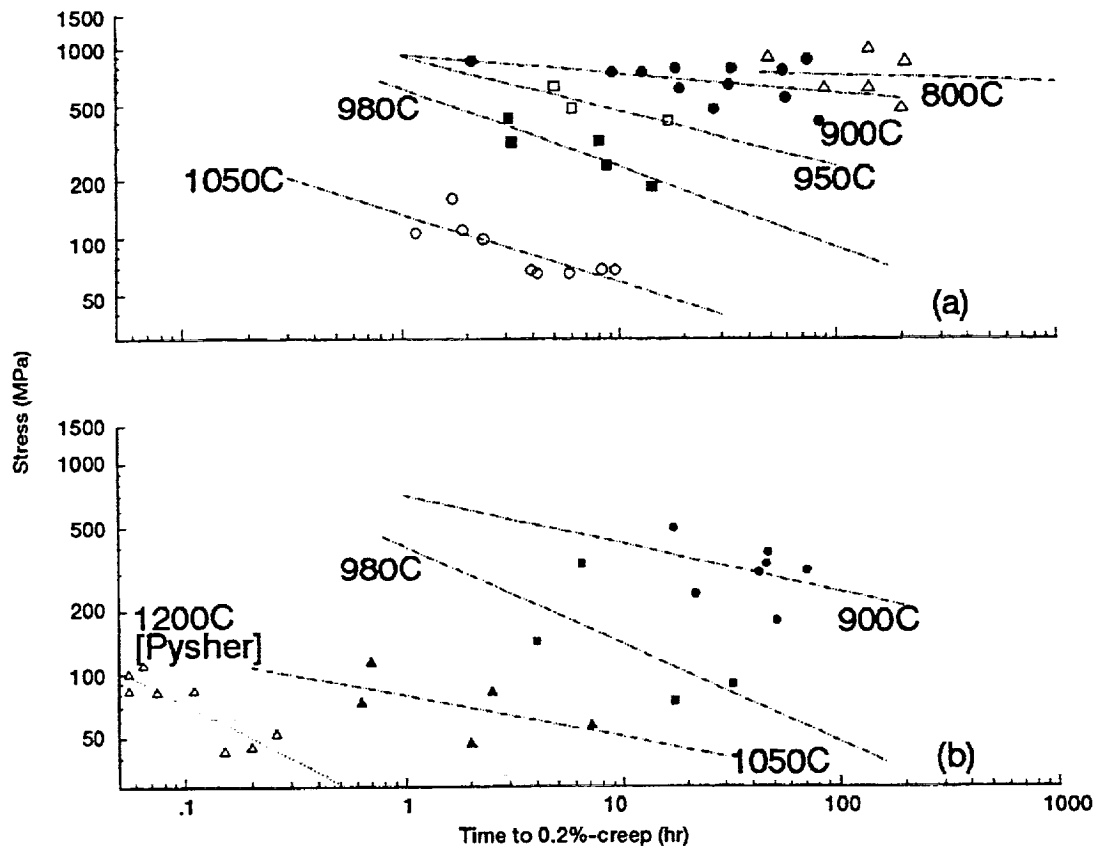


Figure 6.—0.2 percent creep strength of (a) Nextel 610 and (b) Fiber FP.

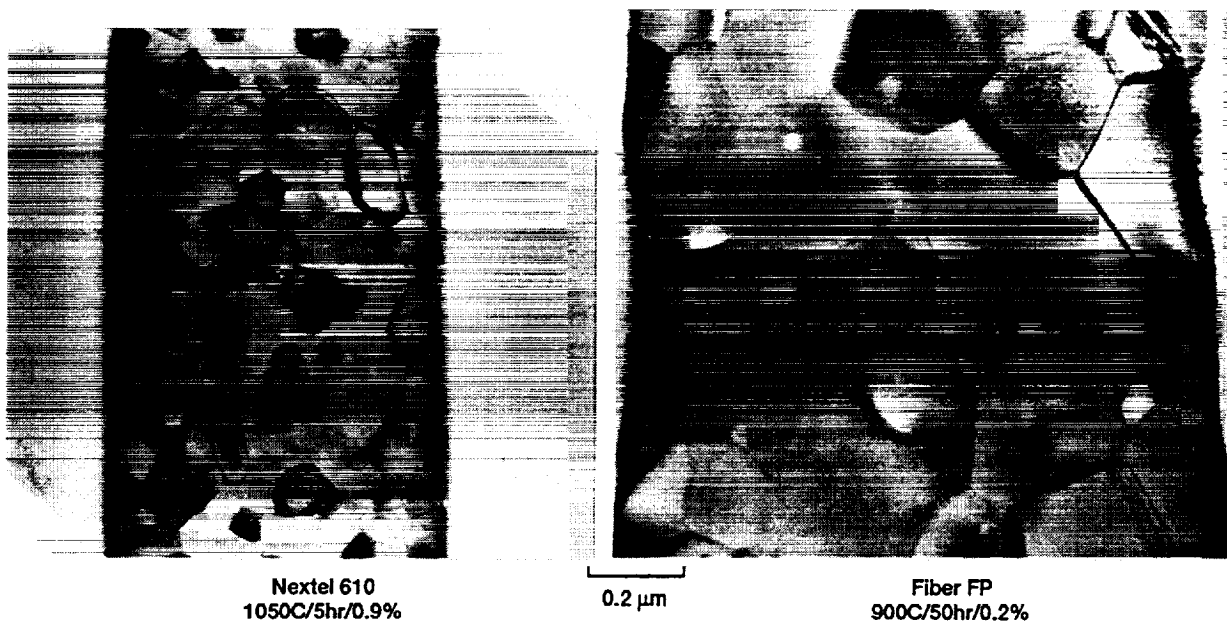


Figure 7.—TEM micrographs of crept polycrystalline alumina fibers.

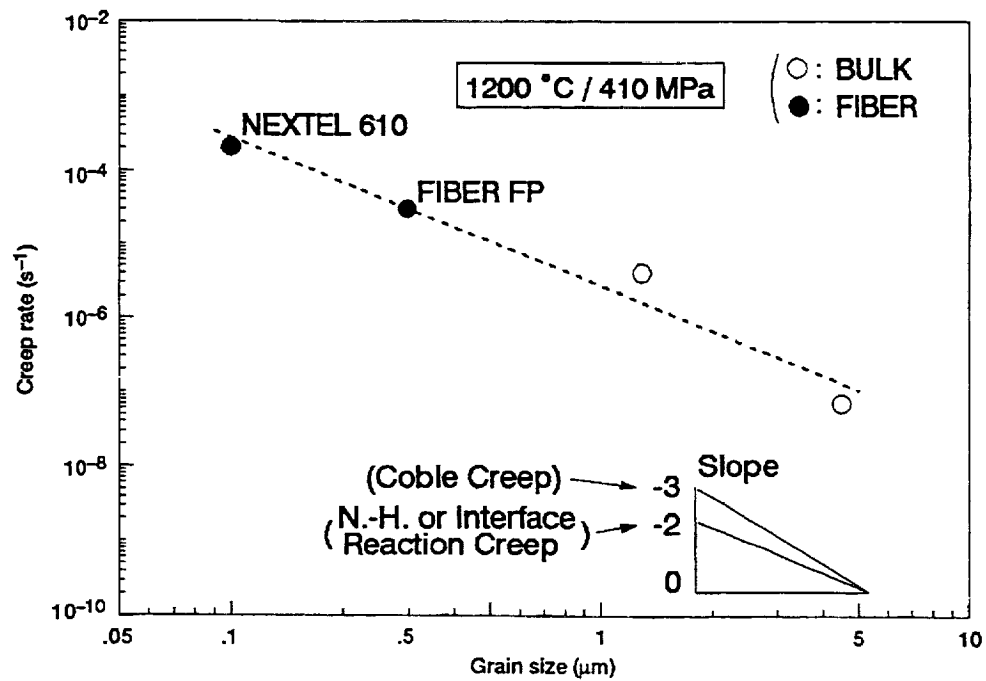


Figure 8.—Effect of grain size on steady-state creep rate of polycrystalline alumina at 1200 °C.

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